



**PATENT APPLICATION**

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re the Application of

Isao OTA et al.

Group Art Unit: 1793

Application No.: 10/678,093

Examiner: M. MARCHESCHI

Filed: October 6, 2003

Docket No.: 111398.01

For: ABRASIVE COMPOUND FOR GLASS HARD DISK PLATTER

**DECLARATION UNDER 37 C.F.R. §1.132**

I, Isao OTA, a citizen of Japan, hereby declare and state:

1. I have a degree in Applied Chemistry which was conferred upon me by School of Engineering of Graduate School of Nagoya University in Nagoya, Japan in 1979.
2. I have been employed by Nissan Chemical Industries, Ltd. since 1979 and I have had a total of 30 years of work and research experience in inorganic oxide fine particles.
3. I am a named inventor in the above-captioned patent application.

4. I and/or those under my direct supervision and control have conducted the following tests: Comparative Test.

(1) Preparation of Sol to be tested

A 500-liter glass-lining reactor was charged with 44.3 kg of pure water and 94.8 kg of aqueous 25% ammonia solution corresponding to  $\text{NH}_3/\text{Ce}^{3+} = 6$  (molar ratio), and 3  $\text{Nm}^3/\text{hour}$  of nitrogen gas was blown therein through a resin nozzle while keeping the liquid temperature at 30°C. Then, 508.0 kg of an aqueous cerium (III) nitrate solution, having a concentration of 7.84 wt% in terms of  $\text{CeO}_2$ , was gradually added thereto over 30 minutes to obtain a suspension of cerium (III) hydroxide. The temperature of the suspension was then elevated to 85°C. Thereafter, the gas to be blown through the resin nozzle was switched from nitrogen to 4  $\text{Nm}^3/\text{hour}$  of air, and the oxidation reaction for converting cerium (III) to cerium (IV) was started. The oxidation reaction was completed in 5 hours. The liquid obtained after the completion of the oxidation reaction was returned to room temperature to obtain a reaction mixture containing white fine particles have a pH = 9.2.

The reaction mixture was washed using a rotary filter press (manufactured by Kotobuki Giken Co., Ltd.) to obtain 172 kg of a white slurry having a solid content of 23.2 wt%, pH = 9.1 and an electric conductivity of 40  $\mu\text{S}/\text{cm}$ .

The observation of the washed slurry with Transmission Electron Microscopy (TEM) revealed that the slurry contained particles from 40 to 80 nm. Furthermore, powder X-ray diffraction measurement of the product by drying the resulting fine particles revealed that the particles contained 99.5% cerium in terms of oxides in the total rare earth elements in the abrasive, and that the particles had characteristic peaks that coincided with the characteristic peaks of crystalline cerium (IV) oxide of cubic crystal form having a diffraction pattern with main peaks at diffraction angles  $2\theta = 28.6^\circ$ ,  $47.5^\circ$ , and  $56.4^\circ$  and described in ASTM Card

No. 34-394.

In addition, the specific surface area value of the cerium (IV) oxide particles, measured by a gas adsorption method (BET method), was  $30 \text{ m}^2/\text{g}$ , and the particle size was 28 nm when calculated from the specific surface area by the gas adsorption.

Further, the obtained particles were measured for average secondary particle size using a centrifugal particle size distribution measuring apparatus (CAPA-700, manufactured by Horiba Seisakusho Co., Ltd.). As a result, the particles were revealed to have an average secondary particle size of  $0.40 \text{ }\mu\text{m}$ . To 110 kg of the washed slurry, 10 wt% of nitric acid aqueous solution was added so as to become a molar ratio of  $(\text{HNO}_3)/(\text{CeO}_2)$  of 0.5, and the concentration was adjusted with pure water to obtain 117 kg of an acid sol having a solid content of 20.1 wt%,  $\text{pH} = 4.8$ , an electric conductivity of  $39 \text{ }\mu\text{S}/\text{cm}$ , and a viscosity of  $1.9 \text{ mPa}\cdot\text{s}$ .

350 g of the acid sol and 1217 g of zirconia beads of  $\phi 0.5 \text{ mm}$  were charged in a sand grinder having a volume of 700 ml, and ground by rotating the stirring blade at 1000 rpm for 3 hours. The resulting sol had a solid content of 21.1 wt%,  $\text{pH} = 5.1$ , an electric conductivity of  $1500 \text{ }\mu\text{S}/\text{cm}$ , and an average secondary particle size measured with a centrifugal particle size distribution measuring apparatus (CAPA-700, manufactured by Horiba Seisakusho Co., Ltd.) of  $0.080 \text{ }\mu\text{m}$ . A sol having  $\text{CeO}_2$  concentration of 5 wt% was prepared by adding pure water to the sol obtained above.

## (2) Polishing Test of a Glass Hard Disk

As a glass hard disk, a 3.5-inch aluminosilicate reinforced glass substrate composed of

77.9 wt% of SiO<sub>2</sub>, 17.3 wt% of Al<sub>2</sub>O<sub>3</sub>, and 4.8 wt% of ZnO was used. The substrate was subjected to primary polishing and had an average surface roughness of 7.3 Angstroms (Å).

An artificial leather-type polyurethane abrasive cloth (POLITEX DG™, 38 φmm, manufactured by Speedfan Co.) was applied to the platen of a LAP MASTER LM-15 grinding machine (manufactured by LAP MASTER Co.) and polishing was performed by pressing the surface to be polished of the substrate against the abrasive cloth under a load of 11 kPa. The number of rotations of the press platen was 45 revolutions per minute and the amount of the abrasive slurry fed was 10 ml/minute. After the polishing, the product obtained was taken out, washed with purified water and then dried. From a decrease in weight, the polishing speed was obtained. The average surface roughness (Ra) of the polished surface and the ratio polishing speed to average surface roughness obtained were as follows.

Polishing Speed (nm/minute)	Average Surface Roughness (Å)	Ratio (Polishing Speed/Average Surface Roughness · minute)
10	2.5	40

The results above, when compared to the results found in Table 1 of the present specification, show that an abrasive composed of cerium oxide having an average secondary particle size of 0.08 μm has a very slow polishing speed and a low ratio of polishing speed/average surface roughness.

I hereby declare that all statements made herein of my own knowledge are true, and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and

the like so made are punishable by fine and/or imprisonment under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing therefrom.

Date: Feb 13, 2009

Isao Ota  
DECLARANT'S NAME (TYPED)  
Isao OTA